Pilot Testing of Mercury Oxidation Catalysts for Upstream of Wet FGD Systems

Quarterly Technical Progress Report

April 1, 2005 – June 30, 2005

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ABSTRACT

This document summarizes progress on Cooperative Agreement DE-FC26-01NT41185, "Pilot Testing of Mercury Oxidation Catalysts for Upstream of Wet FGD Systems," during the time-period April 1, 2005 through June 30, 2005. The objective of this project is to demonstrate at pilot scale the use of solid honeycomb catalysts to promote the oxidation of elemental mercury in the flue gas from coal combustion. The project is being co-funded by the U.S. DOE National Energy Technology Laboratory under Cooperative Agreement DE-FC26-01NT41185. EPRI, Great River Energy (GRE), and City Public Service (CPS) of San Antonio are project cofunders. URS Group is the prime contractor.

The mercury control process under development uses catalyst materials applied to honeycomb substrates to promote the oxidation of elemental mercury in the flue gas from coal-fired power plants that have wet lime or limestone flue gas desulfurization (FGD) systems. Oxidized mercury is removed in the wet FGD absorbers and collected with the byproducts from the FGD system. The current project is testing previously identified catalyst materials at pilot scale to provide engineering data for future full-scale designs. The pilot-scale tests are being conducted for 14 months or longer at each of two sites to provide longer-term catalyst life data.

This is the fifteenth, and final full reporting period for the subject Cooperative Agreement. During this period, only the second pilot unit, at CPS' Spruce Plant, was operated. Operation of the first pilot unit at the GRE Coal Creek site was concluded in 2004. That pilot unit was shipped to TXU Generation Company LP's Monticello Steam Electric Station, for mercury oxidation catalyst testing as part of NETL project DE-FC26-04NT41992.

For the second pilot unit, at Spruce Plant, one catalyst activity measurement trip was conducted in April. These results are discussed in this report. Catalyst regeneration tests were conducted in late April, and the activity of the regenerated catalysts was measured in early May. These results are also discussed in the report. At the conclusion of the activity measurements on the regenerated catalysts, the oxidation catalyst pilot unit was shut down. Later in May, it was disconnected from the ductwork at Spruce plant and shipped to URS' Austin, Texas offices for minor repairs and refurbishment. Early in the next quarter, the pilot unit will be shipped to Southern Company's Georgia Power Plant Yates for mercury oxidation catalyst testing as part of NETL project DE-FC26-04NT41992.

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INTRODUCTION

This document is the quarterly Technical Progress Report for the project "Pilot Testing of Mercury Oxidation Catalysts for Upstream of Wet FGD Systems," for the time-period April 1, 2005 through June 30, 2005. The objective of this project is to demonstrate at pilot scale the use of solid honeycomb catalysts to promote the oxidation of elemental mercury in the flue gas from coal combustion. The project is being co-funded by the U.S. DOE National Energy Technology Laboratory under Cooperative Agreement DE-FC26-01NT41185. EPRI, Great River Energy (GRE) and City Public Service (CPS) of San Antonio are project co-funders. URS Group is the prime contractor.

The mercury control process under development uses catalyst materials applied to honeycomb substrates to promote the oxidation of elemental mercury in the flue gas from coal-fired power plants that have wet lime or limestone flue gas desulfurization (FGD) systems. The oxidizing species are already present in the flue gas, and may include chlorine, hydrochloric acid (HCl), oxygen and/or other species. Oxidized mercury is removed in the wet FGD absorbers and is captured with the FGD byproducts. The objective of this project is to test previously identified catalyst materials at pilot scale to provide engineering data for future full-scale designs. The pilot-scale tests are being conducted for 14 months or longer at each of the two sites to provide longer-term catalyst life data. After completing the project, it is expected that sufficient full-scale test data will be available to design and implement demonstration-scale installations of the catalytic mercury oxidation technology.

The two utility team members are providing co-funding, technical input, and host sites for testing as part of this project. GRE provided the first test site at their Coal Creek Station (CCS), which fires North Dakota lignite, and CPS is providing the second site at their J.K. Spruce Plant, which fires Powder River Basin (PRB) subbituminous coal. These two host sites each have existing wet FGD systems downstream of high-efficiency particulate control devices, an ESP at CCS and a reverse-gas fabric filter (baghouse) at Spruce.

Testing was completed at the first site in 2004, but continued at the second site, CPS' Spruce Plant into the quarter. In May, the oxidation catalyst pilot unit at Spruce Plant was shut down, disconnected from the host site ductwork, and shipped off site, to be used at Southern Company's Plant Yates as part of NETL project DE-FC26-04NT41992. This progress report discusses results from Spruce during this, the last quarter of operation there.

The remainder of this report is divided into five sections: an Executive Summary followed by a section that describes Experimental procedures, then sections for Results and Discussion, Conclusions, and References.

EXECUTIVE SUMMARY

Summary of Progress

The current reporting period, April 1, 2005 through June 30, 2005, is the fifteenth and final full technical progress reporting period for the project. During this period, there was no testing at the first pilot unit site, at the GRE Coal Creek Station, and no related project efforts. For the second pilot unit at CPS' Spruce Plant, the catalyst pilot unit completed its term of operation during the quarter, and was shut down and removed from the host site. One catalyst activity measurement trip was conducted, in April, and catalyst regeneration tests were conducted in late April/early May.

April catalyst activity results from Spruce showed that the fabric filter outlet flue gas mercury content is still highly oxidized, resulting in relatively low inlet elemental mercury concentrations to the pilot unit (generally around 1 $\mu g/Nm^3$). Also, significant temporal variations in concentration make it difficult to quantify catalyst oxidation activity. To improve the accuracy of the mercury oxidation measurements at low concentrations, two newer mercury SCEMs with more sensitive atomic absorption detectors were used to simultaneously monitor the pilot inlet and catalyst outlet locations.

The results from April show that the palladium and gold catalysts each had lost significant activity for elemental mercury oxidation since the last high-quality activity measurements in October 2004. Both were achieving only approximately 50% elemental mercury oxidation. The SCR catalyst activity was reduced to where it was achieving only about 30% elemental mercury oxidation. The activity of the carbon-based catalyst was not measured in April due to an oversight in setting up the outlet mercury SCEM measurement cycle, but results from February showed that this catalyst was achieving less than 10% elemental mercury oxidation.

After these measurements were completed, thermal regeneration of these catalysts was attempted in an air atmosphere. The maximum regeneration temperature achieved as measured at the outlet of the catalyst chambers was only about 350°F. In spite of not achieving a higher temperature, all four catalysts were observed to markedly improve in activity for oxidation of elemental mercury. The palladium and gold catalysts improved to about 80% elemental mercury oxidation, the SCR catalyst improved to nearly 70% oxidation, and the carbon catalyst improved to nearly 60% oxidation.

Problems Encountered

The were no significant problems encountered during the reporting period beyond technical issues discussed in Section 4 of this report.

Plans for Next Reporting Period

The project period of performance ended at the end of the current reporting period (June 30, 2005), so no testing is scheduled at either of the two sites. Both pilot units have been shut down and moved to new sites as part of the 41992 project. The only remaining project effort is submittal of the final report.

EXPERIMENTAL

The work described in this technical progress report was conducted using an elemental mercury catalyst oxidation pilot unit (8000 acfm of flue gas treated) located at CPS' Spruce Plant in San Antonio, Texas. The pilot unit has four separate compartments that allow four different catalysts to treat flue gas from downstream of the host plant's particulate control device and upstream of its FGD system. Details of the pilot unit design, construction, catalyst preparation and pilot unit operation have been discussed in previous quarterly technical progress reports^{1,2,3,4}.

The activity of these catalysts is being determined by measuring the change in elemental mercury concentration across each catalyst, while ensuring that the total mercury concentrations do not change significantly across the catalyst. These measurements are primarily being conducted using a mercury semi-continuous emissions monitor (SCEM) developed with funding from EPRI. The analyzer has been described in a previous report⁵. Periodically, the analyzer results are being verified by conducting manual flue gas sampling efforts in parallel across each catalyst chamber by the Ontario Hydro method.

RESULTS AND DISCUSSION

This section provides details of technical results for the current reporting period, April 1 through June 30, 2005. The technical results discussed are from operation of the second pilot unit at CPS' Spruce Plant.

Background

The pilot unit was started up at Spruce Plant in late August 2003 and operated with the Pd #1 and gold (Au) catalysts installed for most of the month of September. The host unit came off line for a fall outage the evening of September 26, and the outage continued until October 27, 2003. The two remaining catalysts (SCR and C #6) were installed in the pilot unit and the pilot unit was restarted on November 13, 2003. The unit has operated continuously with all four catalysts on line since then, except for periods of host plant outages. The only extended outage during that time period was a recent outage from mid-February through mid-March of 2005.

Pilot unit inlet and catalyst outlet mercury concentration data were first collected for all four catalysts the week of December 8, 2003. SCEM relative accuracy tests by the Ontario Hydro Method were conducted at the same time. The week of January 5, 2004, two SCEMs were taken to the site and used to measure flue gas total mercury and elemental mercury concentrations at the fabric filter inlet and outlet, and at the wet FGD outlet locations on the host unit. These measurements were made to develop a baseline characterization of host unit flue gas mercury conditions prior to rebagging the fabric filter with new bags. The rebagging began on January 12, 2004. Routine catalyst activity measurements by Hg SCEM were made on February 13, 2004, after 11 of the 14 compartments in the west fabric filter (directly upstream of the catalyst pilot unit) had been rebagged. The rebagging was completed at the end of February 2004. Subsequent catalyst activity measurements were made by SCEM in May and August 2004. In October 2004, catalyst activity measurements were made across all four catalysts by mercury SCEM and by the Ontario Hydro Method.

A catalyst activity measurement trip was conducted during the previous quarter, in February, with measurements by mercury SCEM. This measurement trip was unsuccessful, due to a variety of measurement anomalies that were exacerbated by high mercury oxidation across the reverse gas fabric filter on the host unit, and variable total mercury concentrations. The latter two effects tended to make the elemental mercury concentration in the catalyst pilot unit inlet flue gas very low and quite variable. The February measurement trip was intended to be the final trip before the catalyst pilot unit was shut down. However, because the desired measurements of final catalyst activity were not successfully completed, it was decided to wait until after the Spruce Plant outage was completed, to make one more attempt at measuring "end of test" catalyst activity. These measurements were made during the current quarter, in April 2005.

Catalyst Pressure Drop Results

The pressure drop across the four catalyst chambers at Spruce remained nearly constant below 1 in. H₂O during the current quarter. In over 17 months of operation, sonic horns were not required

to prevent fly ash buildup, most likely because a high-efficiency reverse-gas fabric filter is used for particulate control at this site. The use of a fabric filter results in a low dust loading in the pilot unit inlet flue gas, and a dust loading that has less residual electrostatic charge than would flue gas downstream of an ESP.

Catalyst Activity Results

One catalyst measurement trip was conducted during the quarter, April 20-22, 2005. The pilot unit had been re-started on flue gas operation on March 29 following the Spruce Plant outage, so the catalyst had seen over three weeks of flue gas service since being off line for a month.

Because of the measurement issues in February, a number of extra quality assurance/quality control measures were implemented during the April trip. One was to sample the catalyst pilot unit inlet flue gas via two separate sample trains. One train used the normal, permanently installed tubing manifold, solenoid valves, inertial gas separator (IGS) filter and blower that have been used to sample the pilot unit inlet and the outlets of each of the four catalysts throughout the project duration. The other train used a temporary piping, IGS and blower installation on a 4-in. port on the pilot unit inlet duct. The two measurement trains tracked changes in inlet total and elemental mercury concentrations over time very well, but the absolute values measured did not always agree well. The measured inlet flue gas elemental mercury concentrations were relatively low, though $(1-2 \, \mu g/Nm^3)$, by either sampling system, which exacerbated the difficulty of getting good agreement between two independent measurements.

For this reason, it was decided to report the catalyst oxidation performance based on the train sampling the inlet flue gas through the permanent manifold assembly, since the catalyst outlet values were also measured by this train. A negative aspect of this decision is that it did not allow simultaneous measurement of the catalyst inlet and outlet concentrations, since a common manifold is used. However, the negative aspects of not being able to sample simultaneously were minimized by measuring inlet flue gas concentrations immediately before and after sampling each catalyst outlet.

The results of these "end of test" activity measurements are summarized in Table 1. Note that because of an oversight in setting up the sample solenoid sequencing during this time period, no data were collected for the performance of the C #6 catalyst. Instead, data collected in February 2005 that appeared to have been of acceptable quality are shown in the table. The data show that the Pd and Au catalysts were each achieving approximately 50% Hg⁰ oxidation, while the SCR catalyst was achieving about 30%. The February data show that the C #6 catalyst was achieving less than 10% oxidation.

Because considerable effort was expended trying to get high-quality data for catalyst inlet and outlet elemental mercury concentrations, only two of the catalysts were checked for total mercury breakthrough (Pd #1 and Au). These data are summarized in Table 2.

Table 1. April 2005 "End of Test" Catalyst Oxidation Activity Data

Catalyst	Catalyst Inlet Hg ⁰ , (mg/Nm ³ corrected to 3% O ₂)*	Catalyst Outlet Hg ⁰ (mg/Nm ³ corrected to 3% O ₂)*	Observed Hg ⁰ Oxidation Across Catalyst (%)
Pd #1	1.32	0.64	51
C #6	1.26**	1.18**	6**
Au	1.48	0.78	47
SCR	0.80	0.56	29

^{*}Note $-1.0 \mu g/Nm^3$ at 3% O_2 equals $0.67 lb/10^{12}$ Btu heat input

Table 2. April 2005 "End of Test" Catalyst Mercury Breakthrough Data

Catalyst	Catalyst Inlet Total Hg (mg/Nm³ corrected to 3% O ₂)*	Catalyst Outlet Total Hg (mg/Nm³ corrected to 3% O ₂)*	Observed Total Hg Breakthrough Across Catalyst (%)
Pd #1	11.0	12.7	116
C #6	**	**	**
Au	11.0	10.2	93
SCR	**	**	**

^{*} Note $-1.0~\mu\text{g/Nm}^3$ at 3% O_2 equals 0.67 lb/ 10^{12} Btu heat input

It was expected that after the unit outage, and exposure of the catalysts to ambient air, they would have desorbed some mercury and might have taken a few days to re-equilibrate. The results in Table 2 show that these two catalysts had achieved a high percentage mercury breakthrough in the three weeks they had been in service following the unit outage, indicating that they were no longer adsorbing an appreciable amount of mercury from the inlet flue gas.

The activity data were used to generate a plot of measured catalyst activity versus time for the four catalysts in service at Spruce. This plot is shown in Figure 1. The data do not show a consistent trend for activity versus time in flue gas service. Only for the Pd #1 catalyst is there an apparent linear decrease in activity over time, for the time period May 2004 through April 2005 (shown as a dashed line on the figure). Even for this catalyst, the data prior to May 2004 do not fit this apparent linear relationship, and there are even prior data that showed lower activity than the "end-of-test" data.

For the gold and C #6 catalysts, the end-of-test data show considerably lower performance than might have been expected based on the trends seen in the data collected between May and October 2004. The SCR catalyst end-of-test data were consistent with a significant drop in activity seen in the October 2004 data.

^{**}Data collected February 2005

^{**}Not measured

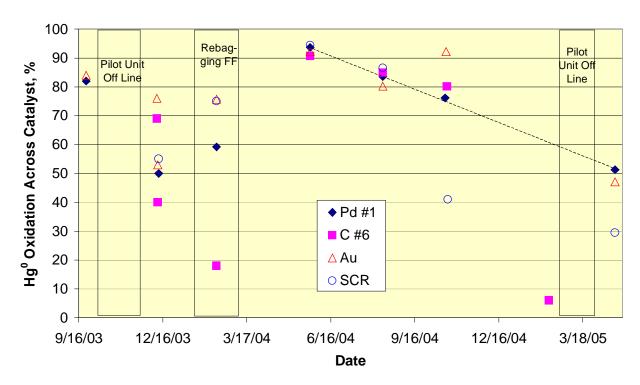


Figure 1. Spruce Catalyst Activity Date vs. Time

Given the difficulties in measuring catalyst performance in February and April 2005, and the extremely low and variable catalyst inlet elemental mercury concentrations, there was even some question as to whether these end-of-test performance values truly represent a loss of catalyst activity, or just that the catalyst outlet elemental mercury concentrations were too low to reliably measure by SCEM. Catalyst thermal regeneration tests were conducted the week of April 25, and the results of those tests are presented below. They indicate that the end of test values do represent activity losses that could be recovered by thermal regeneration.

Catalyst Regeneration Test Results

Catalyst thermal regeneration tests were conducted at Spruce the week of April 25. The tests were conducted by heating plant air with a 36-kW electric heater, then introducing the heated air through an existing port in the catalyst inlet transition (a port intended for sonic horns that were never required). Each catalyst was regenerated individually, and the intent was to isolate the catalyst chamber so that only heated air flowed over the catalyst. However, in actual practice there was some flue gas leakage through the catalysts as well. Each catalyst regeneration test was run from one morning to the next, with nearly 24 hours of elapsed time during the regeneration.

The regenerator was rebuilt from what was used previously at Coal Creek Station, which had failed due to corrosion and wear and tear from being moved several times. The rebuilt regenerator was made to sit beside the catalyst pilot unit rather than bolt directly to the port on the catalyst inlet box. While this reduced wear and tear on the regenerator associated with moving it, the fact that the heated air had to be ducted from the regenerator to the port on the catalyst box resulted in heat losses that limited the regeneration temperature achieved. The maximum regeneration temperature achieved at the catalyst outlet thermocouple for the Spruce

regeneration tests was approximately 350 to 360°F, whereas in the Coal Creek regeneration tests the maximum temperatures were between 400 and 410°F. However, the regeneration gas flow rates were higher in the Spruce regeneration tests (400 to 500 acfm vs. 280 acfm).

In spite of the lower temperatures achieved, all four catalysts appeared significantly improve in elemental mercury oxidation activity after regeneration. Trend plots showing the regeneration temperature, regeneration air flow, and total mercury concentrations measured in the catalyst outlet regeneration air are shown in Figures 2 through 5. Mercury concentrations in the outlet air flow were measured as an indicator for how effectively species adsorbed from the flue gas over time were desorbed during regenerations. Of course, the actual species that cause a loss of activity in the catalysts have not been identified to date, and it is not known what conditions are required to desorb these species relative to what is required to desorb mercury.

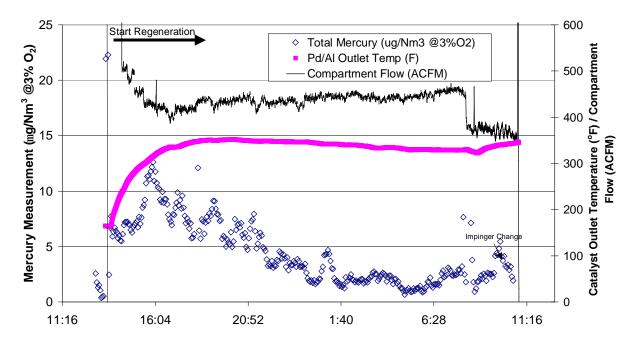


Figure 2. Trend Plot for Regeneration of the Pd #1 Catalyst

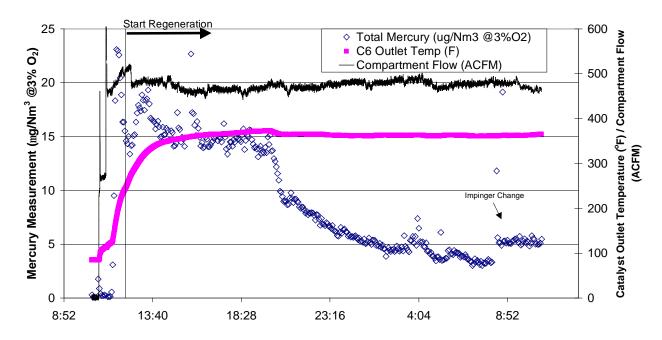


Figure 3. Trend Plot for Regeneration of the C #6 Catalyst

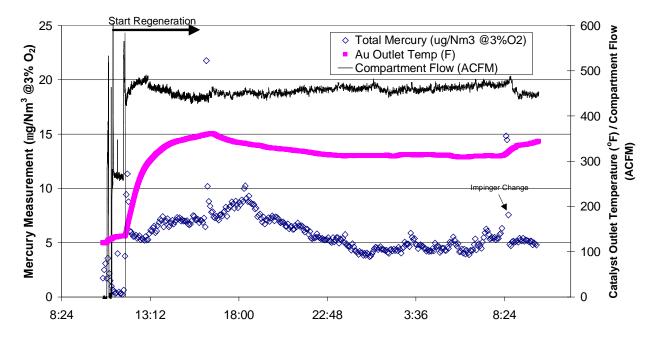


Figure 4. Trend Plot for Regeneration of the Au Catalyst

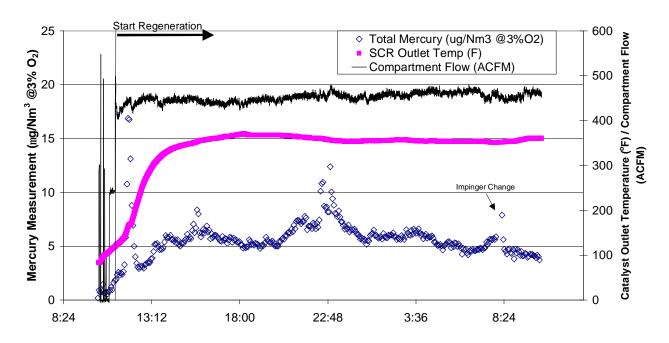


Figure 5. Trend Plot for Regeneration of the SCR Catalyst

The results plotted in the figures show that only for the Pd catalyst did the outlet mercury concentration peak and then return to near zero concentration (\sim 2 µg/Nm³ or less). The C #6 and Au catalyst outlet regeneration air mercury concentrations peaked early in the regeneration, but stayed near 5 µg/Nm³ after the overnight regeneration. The outlet air from the SCR catalyst regeneration slowly increased to about 5-6 µg/Nm³ at the beginning of the regeneration and stayed near that value for the whole regeneration period, other than one brief increase about 12 hours into the regeneration. Based on the mercury concentration data alone, we would speculate that the Pd catalyst was most effectively regenerated at this temperature and duration. As discussed below, the Pd catalyst also proved to be the most active after regeneration by a small margin.

The catalyst activity data for the regenerated catalysts are shown in Table 3. As mentioned above, the Pd catalyst was the most active after regeneration, increasing from 51% elemental mercury oxidation prior to regeneration to 84% afterward. The gold catalyst saw nearly as great an improvement, going from 47% oxidation to 78%. The other two catalysts, which were considerably less active prior to regeneration, also saw substantial improvements in catalyst activity. The C #6 catalyst improved from less than 10% oxidation to 56%, while the SCR catalyst improved from 29% to 66%.

The regenerated catalysts were operated in their normal flue gas environment over a weekend prior to making these activity measurements, the week of May 2nd. The total mercury concentration data summarized in Table 4 show that all of the catalysts were close to mercury adsorption equilibrium when these performance data were collected.

Table 3. May 2005 Post-regeneration Catalyst Oxidation Activity Data

	Catalyst Inlet Hg ⁰	Catalyst Outlet Hg ⁰	Observed Hg ⁰
	(mg/Nm ³ corrected to	(mg/Nm ³ corrected to	Oxidation Across
Catalyst	3% O ₂)*	3% O ₂)*	Catalyst (%)
Pd #1	1.17	0.19	84
C #6	0.94	0.41	56
Au	0.88	0.19	78
SCR	0.90	0.30	66

^{*}Note – 1.0 $\mu g/Nm^3$ at 3% O_2 equals 0.67 $lb/10^{12}$ Btu heat input

Table 4. April 2005 "End of Test" Catalyst Mercury Breakthrough Data

Catalyst	Catalyst Inlet Total Hg (mg/Nm³ corrected to 3% O2)*	Catalyst Outlet Total Hg (mg/Nm³ corrected to 3% O2)*	Observed Total Hg Breakthrough Across Catalyst (%)
Pd #1 (a.m. data)	5.3	5.9	110
Pd #1 (p.m. data)	10.1	9.7	96
C #6	10.4	8.9	85
Au	10.6	10.3	98
SCR	10.8	10.2	94

^{*}Note $-1.0 \ \mu g/Nm^3$ at 3% O_2 equals $0.67 \ lb/10^{12}$ Btu heat input

CONCLUSION

At the Spruce site, the fabric filter upstream of the pilot unit has had two implications on the pilot testing. One is that is does not appear that sonic horns are required to keep fly ash from accumulating within the catalyst cells. The other implication is that the fabric filter oxidizes a high percentage of the elemental mercury in the air heater outlet flue gas, so the inlet gas to the pilot unit contains relatively low elemental mercury concentrations (typically <1 to $4 \mu g/Nm^3$). This makes evaluation of catalyst performance difficult, as it is difficult to quantify catalyst outlet flue gas elemental mercury concentrations that are well below $1 \mu g/Nm^3$.

The catalyst measurements conducted during the quarter represented the end-of-test performance for the four catalysts in service at Spruce Plant. All four showed significantly lower mercury oxidation activity than they had during the last successful measurements in October 2004. No firm conclusion can be drawn from the activity versus time data regarding catalyst deactivation rates. While it would be possible to assume a linear decrease starting with the initial catalyst activity at the beginning of the test and the final activity measured in April (February for C #6 catalyst), such a relationship would not fit the measured data in between for any of the four catalysts. This is particularly true for the C #6 and SCR catalysts, where the initial activity measurements were made in December 2003 and showed relatively low activity, but later measurements in 2004 showed significantly higher activity for both.

It is clear, though, that all four catalysts experienced a significant reduction in activity for elemental mercury oxidation by April 2005, as all four showed substantial improvements in activity after thermal regeneration at approximately 350°F for 24 hours. More work is needed to optimize regeneration conditions (temperature, duration, and air flow rate), which may be different for each catalyst type. Furthermore, data should be collected to determine the deactivation rates for regenerated catalysts compared to new catalysts.

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